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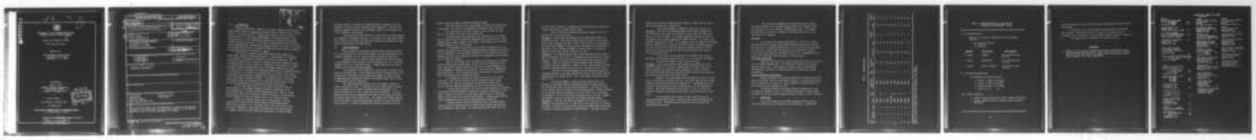

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DEVELOPMENT OF HIGH TEMPERATURE RESISTANT MATERIALS FOR USE IN NAVAL ORDNANCE

Contract No. N00017-73-C-4306

Fifth Quarterly Report

Submitted to:

Naval Ordnance Systems Command
Department of the Navy
Washington, D. C. 20360

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The scale-up 18" furnace was constructed and is currently being subjected to bake-out and heat-up tests. A closed water system is now in operation permitting furnace operation independent of city water.

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1.0 INTRODUCTION

Pyrostrand® graphite composite is a carbon filament reinforced pyrolytic graphite formed by simultaneous pyrolytic graphite deposition and filament addition. A previous program[1] demonstrated attractive erosion resistant characteristics for rocket nozzle applications. Additional features of this concept include: (1) the simultaneous deposition of carbides (when desired) to modify the matrix structure, (2) the use of filamentary materials of various textile configurations and mechanical properties and (3) the control of reinforcement placement and content for increased strength and greater erosion resistance in very-high-temperature environments.

The pyrolytic graphite matrix has inherently high resistance to erosion under rocket motor firing conditions. A disadvantage of the material is its tendency to delaminate at moderate or excessive thicknesses. This delamination tendency can be reduced by causing the pyrolytic graphite to grow from surfaces other than the substrate, a filamentary reinforcement for example. The high performance graphite yarns have a very high strength/density ratio and thereby offer the potential for producing composites with a significant degree of reinforcement. In contrast to most materials, the strength of both major components of these composites increases with temperature up to near the sublimation temperature of graphite (6,600°F). The graphite fiber reinforcement sufficiently reduces the anisotropy of the pyrolytic graphite matrix so that freedom from delaminations of the pyrolytic graphite is achieved.

Rocket motor nozzle tests in 1-inch throat sizes have shown the applicability of these graphite composite materials (Pyrostrand) to the nozzle conditions of a typical advanced propulsion system. Results to date indicate that, under test conditions of 6500°F and 1000 psi, the Pyrostrand graphite composite erodes at an average rate of 2 ± 1 mils per second.

Based on the expected erosion rate of approximately 2 mils per second, plus the high potential hoop strength, the Pyrostrand graphite composite nozzle appears to be a desirable throat insert material for these rocket motor conditions. Other nozzle candidates have major disadvantages. For example, tungsten has high weight and vulnerability to nuclear damage,

pyrolytic graphite coatings have a maximum thickness capability and bulk graphites or ablative carbon composites erods excessively. The Pyrostrand graphite composite materials are structurally capable of self support, due to the filamentary reinforcement.

Past efforts have been limited by furnace size and a small capacity for handling the filamentary material. The results were sufficiently promising to indicate that further work should be conducted to scale-up the equipment for the production of larger pieces.

2.0 WORK ACCOMPLISHED

Work accomplished during this reporting period related primarily to (1) the construction of the 18-inch furnace, (2) the installation of the accessory components, and (3) furnace bakc-out and heat-up studies.

Construction of the 18-inch furnace involved four major sections: (1) the atmosphere control chamber; (2) the induction coil; (3) the furnace assembly and (4) the closed water system. Company funds were used for the purchase of the above mentioned equipment.

The atmosphere control chamber is a vacuum tank, 72 inches in diameter and 60 inches high. It was constructed from 304 stainless steel in two sections split horizontally and provided with flanges for vacuum sealing. A tongue-and-groove design was provided for operations under either vacuum or differential pressure conditions.

The tongue-and-groove surfaces had to be slightly reworked to effect a satisfactory seal. In addition, the solid rubber gasket provided with the chamber was of irregular thickness and incompressable in the snug fitting groove, such that a vacuum seal was not possible. The solid gasket was removed and a compressable flat silicone foam ring was bonded to the outside fing of the top flange area. The tongue on the flange prevents the silicone gasket from moving inward when subjected to less than atmospheric pressure. A pressure sensing device has been installed within the

furnace to warn of sudden, inadvertent pressure changes.

The lower section of the vacuum chamber contains three observation or yarn ports. All utility connections pass through the lower tank wall.

The lower half of the tank is fixed in place and only the upper tank lid is removed for access to the furnace area.

New concepts were incorporated in the construction of the furnace with the furnace structure based upon experience with the 8-inch unit. The maximum furnace chamber diameter is 19 inches. This diameter will accomodate hardware diameters up to 12-1/2 inches.

A cooling plate on the bottom of the furnace is bolted to the coil assembly and supports the susceptor, winding assembly and lampblack insulation. It is now possible to lift the entire furnace out of the bottom tank section; however, there are line connections that must be disconnected at the accessory port to remove the furnace.

Control flow meters,as well as visual flow indicators were attached to each injector water cooling line. In the event of an inadvertent water leak, that specific defective system can be shut off without damaging the other components by complete water shut-off. All outlet water temperatures are now monitored. Also, the induction coil cooling water is monitored by sight flow indicators and temperature sensors.

Two independent cooling coil lines are attached to the furnace base plate. Each line is monitored and has a separate control system. In the event of a leak in a cooling line, the defective system can be shut off and the remaining independent line used to cool the base plate.

A closed water system for use with the vaccum furnaces was completed. The system has provided adequate cooling water capacity independent of city water. However, city water is automatically directed into the recycling system in the event of an electrical power failure. Furnace support systems such as plumbing and control pannel installation were completed.

Several check-out tests were performed with the 18-inch furnace. Initial tests were conducted with the furnace under vacuum conditions and all cooling water lines opened. The vacuum leak rate was found to be very

low (0.1" Hg in 5 minutes). Examination of the furnace after vacuum tests revealed no structural damage or water leaks.

Five test runs were performed using power applied to the coil, and each test is described below:

Test Run 1 - Cooling water was supplied by the closed water system at a pressure of 50 psi. The furnace was electrically powered for 20 seconds at zero power settings (324 volts, 43KW). The coil did not vibrate and was very quiet when energized. After the short 20 second run, the susceptor was found to have been heated slightly, to about 100°F. No structural damage or water leaks resulted form this test run.

Test Run 2 - The furnace was put under vacuum conditions and was found to have an acceptable leak rate of 0.1 inches Hg in 5 mintues with all water lines on. The water flow rate through each injector was determined to be 37 gal/hr. Again the induction coil was energized, under minimum power, to achieve a furnace temperature of 800°F (427°C). Slow heating was accomplished by turning the power on and off over a time span of 6-1/2 hours.

The top of the vacuum chamber was not inductively heated, but the middle portion of the lower chamber wall was warm. Exit water from the induction coil was measured at 68 - 70°F. Exit water from the base plate was 70 - 71°F.

No furnace damage or leaks were found after the test run. However, the hoisting brackets, positioned between the support studs on the cooling coil turns, had been heated resistively. The induced current in the cooling turns was shorted by the hoisting brackets. Excessive heating of the brackets caused carbonizing of the outer layer of the epoxy-glass coil supports. Two of the studs were melted back about one inch. Damaged stud support areas were repaired and the hoisting brackets removed.

Test Run 3 - A silicone foam gasket was bonded to the outer half of the flange on the vacuum tank lid. A leak rate of 0.35 inches Hg in 20 minutes was noted. The furnace was heated for 3 hours at minimum power (on and off) to achieve a measured temperature of 1550°F (843°C). Exit water from the

induction coil reached a maximum of 75°F (power on longer than Test Run 2) and the base plate water reached 80°F.

No structural damage or leaks were found after the test run.

Test Run 4 - Test Run 4 was performed using a continuously energized coil for 5-1/2 hours. The power was increased in eight steps (from 20KW, 326 volts to 95 KW, 470 volts). An average temperature increase of 400°F/hr was measured, with a peak temperature of 2260°F (1240°C). The chamber wall reached a temperature of 325°F. It was apparent that the chamber is suscepting with the induction coil. The exit coil water reached a maximum temperature of 77°F. Water from the injectors reached a maximum temperature of 84°F. Base plate cooling water peaked at 109°F in the secondary line and 99°F in the primary exit line.

A leak rate test was performed on the chamber after the run and found to be the same as before the run, i.e., 0.35 in Hg in 20 minutes.

No structural damage or water leaks were found.

Test Run 5 - This test run was performed using the energized coil for a continuous period of 4-1/4 hours. Maximum power used was 118 KW at 510 volts, resulting in a measured furnace temperature of 2645°F (1232°C). Exit coil water temperature was measured at 88°F; injector water exit water was 96°F; base plate water was 114° and 123°F.

However, the vacuum chamber wall was measured at 400°F, at which time it was decided to terminate the run. The vacuum tank wall was heating because it was suscepting with the inductive coil. Ajax Magnethermic was contacted to explain why the shell is suscepting, as it was designed not to do so. They cannot explain why it suscepts. We plan to resolve the problem of heating by installing cooling coils on the exterior side of the shell.

The epoxy-glass plate around the power leads entering the vacuum tank heated high enough to cause the inner layer to delaminate and the epoxy to char. Cooling coils on the tank wall should prevent this condition from recurring.

The furnace was undamaged and no leaks were found. However, the exhuast chamber liner and upper exhaust chamber were slightly eroded and contained a very thin coating of reddish-brown powder. This powder was identified as iron oxide. Efforts to determine the source for the iron oxide are in progress.

TEST PLAN

In accordance with verbal instructions of 8 June 1973, Atlantic Research has been in contact with Battelle Columbus Laboratories personnel for the purpose of establishing direction for our current Pyrostrand program. The intent is to insure that this program is consistent with the goals of the overall Navy Materials Development effort. Our recommendations for future work, based upon these meetings, are outlined below.

18" Furnace Development

Completion and check-out of the 18" Pyrostrand furnace will continue to be a primary immediate goal. The furnace was installed as an Atlantic Research capital item. Continuation of check-out tests and preliminary windings will be performed to demonstrate that the furance meets design requirements.

Physical Properties Determination

Atlantic Research and Battelle personnel have established both the physical properties tests and a systematic variation of operating parameters for the fabricaiton of test parts. Table 1 defines the desired parts to be fabricated during the remainder of 1973.

The minimum property evaluations desired in evaluating the test specimens are descirbed in Table 2. Property evaluations will most likely be limited to baseline process parameters shown as items 1, 2 and 9 in Table 1.

3.0 FUTURE WORK

The 18-inch furnace will continue to undergo check-out tests, after the cooling coils are attached to the outside wall. Winding tests

TABLE 1. FABRICATION PLAN

Test No.	Quantity	Size	Fiber	Wt % Fiber	Deposition Temperature, F	MTS, oz/hr	Deposition Time,	Turns/in.	Dwell	Rotation, rpm	Wraps	Furnace, in.
1	3	A	WYB85	7	3800	0	15	20	30	4	10	8
2	3	A	WYB85	14	3800	0	15	40	30	8	10	8
3	1	A	Th50	7	3800	0	15	20	30	4	10	8
4	1	A	Th400	7	3800	0	15	20	30	4	10	8
5	1	A	MF2.5 mil	7	3800	0	105	280	75	8	10	8
6	1	A	WYB85	14	3200	1	15	40	30	8	10	8
7	1	A	WYB85	7	3800	0	15	20	30	4	10	18
8	1	A	WYB85	14	3200	1	15	40	30	8	10	18
9	1	B	WYB85	7	3800	0	45	20	0	4	10	18
10	1	A	WYB85	7	3800	0	15	20	30	4	20	18

Size A = 3-in.-ID, nominal 3/16-in. wall thickness, 3-in. length.
Size B = 5.5-in. ID, nominal 3/16-in. wall thickness, 6-in. length.

TABLE 2. MINIMUM PROPERTY DATA REQUIRED FOR EVALUATION OF PYROSTRAND® PROCESS

I. At Room Temperature

Rings are to be used for fabrication of all specimens. Rings are:

ID = 3.00 in./5.500 in.
W = 0.250 in.
t = 3/16 in.

Quantity	Type of Test	Data Measured
5 (ring)	Tensile	Stress-Strain Curve σ_{11}, E_{11}
5 (ring)	Compression	Stress-Strain Curve σ_{11}, E_{11}
5 (ring)	Shear (flexural)	Stress-Strain Curve τ_{12}, G_{12}

II. At Elevated Temperatures

All specimens are 3-in.-diam rings

2	Tensile at 1000 F	Ultimate
2	Tensile at 2000 F	Tensile
2	Tensile at 3000 F	Strength
2	Tensile at R.T.	σ_{11}

III. Thermal Properties

1. Thermal diffusivity from RT to 2500 C (radial direction)
2. Thermal expansion from RT to 2500 C (radial and axial direction)

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will be performed as soon as the furnace has been determined to meet critical requirements.

Utilization of the 8-inch furnace in preparing the 3" ID samples will continue. Fabrication of both 3" ID and 5.5" ID samples will be initiated in the 18" furnace as soon as furnace check-out tests are completed.

Test samples will be submitted for property test data.

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